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May 12, 2006

PATENT APPLICATION
Attorney's Docket No.: 1021.2005-001



Applicant:

Yi Hua Ma, Ivan P. Mardilovich and Erik E. Engwall

Application No.: 10/804,846

Group: 1724

Filed: March 19, 2004

Examiner: Frank M. Lawrence, Jr.

Confirmation No.: 7497

Title: Composite Gas Separation Modules Having Intermediate Porous Metal Layers

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| CERTIFICATE OF MAILING OR TRANSMISSION | |
| I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as First Class Mail in an envelope addressed to Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450, or is being facsimile transmitted to the United States Patent and Trademark Office on: | |
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DECLARATION UNDER 37 C.F.R. § 1.132 OF ERIK E. ENGWALL, Ph.D.

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Erik E. Engwall, Ph.D. of Houston, Texas, declare and state that:

1. I am a co-inventor of the subject matter described in U.S. Serial No. 10/804,846, claiming composite gas separation modules, methods for fabricating a composite gas separation module, methods for selectively separating hydrogen gas from a hydrogen gas-containing gaseous stream, hydrogen gas separators, methods of purifying hydrogen gas, and methods of manufacturing hydrogen gas separators. I also am a co-inventor of the subject matter described in

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U.S. Provisional Application No. 60/457,061, of which the subject application is claiming the benefit under 35 U.S.C. § 119. The subject patent application was filed on behalf of Worcester Polytechnic Institute, 100 Institute Road, Worcester, Massachusetts 01609.

2. I have thoroughly studied the above-identified, subject patent application and its corresponding provisional application, and also studied the Office Action mailed from the U.S. Patent and Trademark Office on February 24, 2006 in the subject application.

3. I have over 15 years of experience in organic and inorganic materials research. I also am a co-author of several publications and have been a participant in presentations directed to characterization and fabrication of specialty materials, including dense gas-selective membranes. A summary of my professional biography appears as Attachment A.

4. Sulfides and oxides are compounds and are not considered to be metals or metallic. The distinction between metals, or metallic materials, from other materials, such as sulfides and oxides, can be found in differences in disposition of valence electrons. All sulfides and oxides have their valence electrons participating in either covalent or ionic bonding. In contrast, the valence electrons of adjacent atoms in metallic substances are de-localized and loosely shared between large numbers of adjacent atoms.

5. The intermediate metal oxide and sulfide layers disclosed in Peachey et al. '708 would not be considered by one skilled in the relevant art to be intermediate metal layers because metal oxides and sulfides are not considered to be metal materials. Therefore, Peachey et al. '708 do not teach an intermediate porous metal layer, as is claimed in the present application.

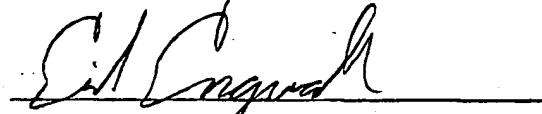
6. One skilled in the relevant art would not consider the oxidized intermediate layer 14 of U.S. 6,152,987 by Ma et al. (Ma et al. '987) to be an intermediate porous metal layer.

7. I hereby acknowledge that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further

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that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



Erik E. Engwall, Ph.D.

5/15/2006

Date

Erik Engwall

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Houston, TX 77082

Phone: (281) 658-2527
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Education

Ph.D., Chemical Engineering, Worcester Polytechnic Institute, Worcester, MA, 2001

- The reduction of the cation exchange capacity (CEC) in natural montmorillonite was explored as a means of controlling the micro pore structure in Al and Zr pillared clays (PILC).
- An ethanol based synthesis was developed for Zr pillaring of hydrophobic CEC reduced clays. The resulting materials had high surface areas ($300 - 350 \text{ m}^2/\text{g}$).
- Zr and Al PILCs were evaluated as supports for CuCl introduced by spontaneous dispersion, to produce π -complexation based adsorbents for ethylene/ethane separation.
- A thermodynamic model was developed to estimate the micropore dimensions of PILCs from small molecule adsorption data.

B.S., Chemical Engineering, Worcester Polytechnic Institute, Worcester, MA, 1987

Professional Experience

Senior Research Engineer, Shell International Exploration and Production, Houston, TX 2005-present

- Pilot and large laboratory scale process and membrane development for MISR (Modular Intensified Steam Reforming). This is a steam reforming process which utilizes a Pd membrane reactor to increase conversion and intensify the process.

Research Assistant Professor, Worcester Polytechnic Institute, Worcester, MA. 2001-2005

- Development of high temperature (500°C) hydrogen separation membranes based on electroless deposition to form Pd or Pd alloy films on porous sintered metal supports.
- Development of inter-metallic diffusion barriers to protect Pd and Pd alloy membranes from support metal diffusion during high temperature (500°C) operation. These technologies resulted in two patent applications.
- Development of a method to close large "defect pores" in sintered metal supports by selective electroless plating of the pore walls in the vicinity of the pore mouth. This yielded thinner Pd membranes with higher hydrogen permeance. This technology resulted in a patent application.
- Development of a model to use room temperature He or N_2 permeance data from membrane supports before plating, to predict the porous support contribution to the pressure drop of a completed membrane module under process conditions.
- Designed and built several automated permeation stations to evaluate the performance of pre-pilot (6" L, 1" OD) Pd and Pd alloy membranes under hydrogen, and in the presence of gas mixtures which simulate the expected gas compositions in contact with the membrane under steam reforming conditions.

SEM Laboratory Manager, Worcester Polytechnic Institute, Worcester, MA. 1999-2000

- Setup and managed the Inorganic Materials Analysis Laboratory (SEM/EDS and XRD) used by Graduate Students and Post Docs in the Chemical Engineering Department.

Attachment A

- Developed operating procedures and implemented a training program to qualify new users.

Adjunct Professor, Worcester Polytechnic Institute, Worcester, MA. 1998-2000

- Lecturer for 3 consecutive years for the WPI undergraduate distillation and separation course (CM-2002), part of the program leading to a B.S. in Chemical Engineering.
- Curriculum Development for a transition to a computer integrated classroom with cooperative and hands-on elements.
- Received consistently high ratings on student evaluations of this course.

Staff Engineer, EIC LABORATORIES, Norwood, MA 1993

- Evaluation porous polymer membranes for use as electrode jacketing materials for lithium batteries.
- Evaluation of fabrication methods for lithium batteries.

Project Engineer, C.R. BARD INC., Tewksbury, MA 1992

- Engineer for a major medical device manufacturer developing arterial fiber optic blood gas sensors.
- Evaluated blood CO₂ sensor performance and stability in the presence of anesthetic gases.
- Evaluated the effects of ethylene oxide sterilization on sensor performance.

Engineer, U.S. ARMY MATERIALS TECHNOLOGY LABORATORY, Watertown, MA 1987-1992

- Member of the Chemical Protection team in the Polymer Research Branch with secret clearance.
- Formulated butyl rubber compounds for use as candidate control/reference materials in glove permeation testing performed by the Product Assurance Directorate.
- Conducted vapor and liquid sorption and permeation testing on rubber glove materials and nylon fabric/butyl rubber composites used for protective garments. These experiments were used in new materials development and in the study of manufacturing defects.
- Designed GC based automated vapor and liquid permeation testing equipment for butyl rubber gloves and other polymer barrier materials.

Skills

Membrane synthesis and testing; Hydrogen separation membranes; Electroless plating of Palladium; Gas and liquid permeation and diffusion in polymers; Volumetric and gravimetric gas adsorption methods; The characterization of porous solids; Inorganic synthesis; X-ray diffraction; Scanning Electron Microscopy; Rubber compounding; Design and troubleshooting of complex experimental equipment; Management of technical teams; Data analysis; Visual Basic for Excel Macros; Technical writing; Excellent communication.

Patents

Yi Hua Ma, Ivan P. Mardilovich, and **Erik E. Engwall**, COMPOSITE GAS SEPARATION MODULES HAVING INTERMEDIATE POROUS METAL LAYERS, Patent Application Publication, US 2004/0237779 A1, Dec. 2, 2004.

Yi Hua Ma, Ivan P. Mardilovich, and **Erik E. Engwall**, METHOD FOR FABRICATING COMPOSITE GAS SEPARATION MODULES, Patent Application Publication, US 2004/0237780 A1, Dec. 2, 2004.

Yi Hua Ma, Ivan P. Mardilovich, and **Erik E. Engwall**, METHOD FOR CURING DEFECTS IN THE FABRICATION OF A COMPOSITE GAS SEPARATION MODULE, Patent Application Publication, US 2004/0244583 A1, Dec. 9, 2004.

Yi Hua Ma, Ivan P. Mardilovich, and **Erik E. Engwall**, COMPOSITE GAS SEPARATION MODULES HAVING HIGH TAMMAN TEMPERATURE INTERMEDIATE LAYERS, Patent Application Publication, US 2004/0244590 A1, Dec. 9, 2004.

Publications and Presentations

Erik Engwall, I.P. Mardilovich, B. Ceylan Akis and Y.H. Ma, Formation and Characterization of Oxide Diffusion Barriers on Porous 316L Stainless Steel Supports used for Composite Pd Membranes, AIChE National Meeting, San Francisco, (2003).

Yi Hua Ma, B. Ceylan Akis, M. Engin Ayturk, Federico Guazzone, **Erik E. Engwall** and Ivan P. Mardilovich, Characterization of Intermetallic Diffusion Barrier and Alloy Formation for Pd/Cu and Pd/Ag Porous Stainless Steel Composite Membranes, Ind. Eng. Chem. Res., v43, 2936-2945, (2004).

E. Engwall, I.P. Mardilovich and Y.H. Ma, Transport Resistance for Oxide Diffusion Barriers on Porous Metal Supports used in Composite Pd and Pd-Alloy Membranes, ACS National Meeting, New Orleans, (2003).

Yi Hua Ma, Ivan P. Mardilovich and **Erik E. Engwall**, Thin Composite Palladium and Palladium/Alloy Membranes for Hydrogen Separations, Annals of the New York Academy of Science, Volume 984, (2003), 346-360.

I.P. Mardilovich, **E. Engwall** and Y.H. Ma, Dependence of hydrogen flux on the pore size and plating surface topology of asymmetric Pd-porous stainless steel membranes, Desalination, 144, (2002), 85-89.

E. Engwall and Y.H. Ma, Characterization of Cu-PILCs prepared by spontaneous dispersion of CuCl on Zr and Al-PILCs, Fundamentals Of Adsorption 7 , K. Kaneko, H. Kanoh and Y. Hanzawa eds., IK International, (2001).

Y.H. Ma, Y. Zhou, R. Poladi and **E. Engwall**, The synthesis and characterization of zeolite A membranes, Separation and Purification Technology, 25, (2001) 235-240.

E. Engwall and Y.H. Ma, Synthesis and Characterization of Al and Zr PILCs from CEC reduced Montmorillonite, Fundamentals Of Adsorption 6, Francis Meunier ed., Elsevier, (1998), 593-598.

H. Gold, Y.H. Ma, **E. Engwall** and P. Mardilovich, Activation of carbon dioxide on activated carbon and removal from carbon through direct electrical resistance heating, NASA Tech Briefs, (1997).

E. Engwall, D.A. Bulpett and D.P. Macaione, Materials characterization in support of butyl rubber control material development, 1990 CRDEC Scientific Conference on Chemical Defense Research, (1990).

E. Engwall, Flooded cell permeation testing of butyl rubber barriers, 1988 CRDEC Scientific Conference on Chemical Defense Research, (1988).

Awards

NASA Space Act Awards for the creative development of a technical innovation for reportable item MFS-26480, Activation of carbon dioxide on activated carbon and removal from carbon through direct electrical resistance heating. September 3, 1997.

DEPARTMENT OF THE ARMY, Official Commendation for the quality and timeliness of work in support of the Product Assurance Directorate (PAD) of CRDEC. August 20, 1991.

DEPARTMENT OF THE ARMY, Official Commendation for efforts to stock, inventory and dispose of hazardous chemicals at the Polymer Research Branch. August 9, 1989.

DEPARTMENT OF THE ARMY, Official Commendation for the development of automated capabilities in carrying out the measurement of the permeation resistance of barrier materials and for work leading to a test program funded by the Product Assurance Directorate at CRDEC. June 27, 1989.